

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:

Yoshimura et al.

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Examiner: SERGENT, RABON A

Title : MEDICAL ADHESIVE

DECLARATION UNDER RULE 132

Honorable Commissioner of Patents and Trademarks,
Alexandria, Virginia 22313-1450

Sir:

I, Tetsuji Yoshimura, a citizen of Japan and having postal mailing address of 4-7-13, Kitayamadai, Konan-shi, Shiga 520-3241 JAPAN, declare and say that:

March 1979, I graduated from Graduate School of Science, Kyoto University, and received a master's degree in chemistry;

From April 1979, up to the present, I have been employed by Sanyo Chemical Industries, Ltd., and engaged in the works of research and development for organic polymers, especially in the field of polyurethane;

In June 2003, I joined the group where the inventors of the present invention belonged;

I am the inventor of the above-identified application and am familiar with the technical field of the present invention;

I have read the Official Action mailed and the references cited therein. I respectfully submit herewith my exact report;

In order to demonstrate the effects of concrete compounds of a fluorine-containing aliphatic diisocyanate (A11) and a phenolic radical scavenger (PRS), I carried out the following experiments.

<Example 16 (modification example 1 of a fluorine-containing aliphatic diisocyanate in Example 1)>

The medical adhesive (P16) of the present invention was obtained by the same method as that in Example 1, except using a mixture of 90 parts of the random co-adduct of ethylene oxide/propylene oxide (b1) obtained in Production Example 1 and 10 parts of the adduct of propylene oxide (b3) obtained in Production Example 3 and 38.3 parts of bis(isocyanatomethyl)perfluoropropane [OCN-CH₂-(CF₂)₃-CH₂-NCO] (the ratio of NCO group/OH group = 2/1) as the fluorine-containing aliphatic polyisocyanate component (A).

As for this (P16), the content of isocyanate groups was 4.1%, the number average molecular weight (Mn) was 5,300, and the content of alkaline metals and/or alkaline earth metals was 0.02 mmol/kg.

<Example 17 (modification example 2 of a fluorine-containing aliphatic diisocyanate in Example 1)>

The medical adhesive (P17) of the present invention was obtained by the same method as that in Example 1, except using a mixture of 90 parts of the random co-adduct of ethylene oxide/propylene oxide (b1) obtained in Production Example 1 and 10 parts of the adduct of propylene oxide (b3) obtained in Production Example 3 and 89.4 parts of bis(isocyanatomethyl)perfluorodecane [OCN-CH₂-(CF₂)₁₀-CH₂-NCO] (the ratio of NCO group/OH group = 2/1) as the fluorine-containing aliphatic polyisocyanate

component (A).

As for this (P17), the content of isocyanate groups was 4.0%, the number average molecular weight (Mn) was 5,800, and the content of alkaline metals and/or alkaline earth metals was 0.03 mmol/kg.

<Example 18 (modification example 1 of a radical scavenger in Example 1)>

The medical adhesive (P18) of the present invention was obtained by the same method as that in Example 1, except that 2,6-di-t-butyl-p-cresol (Antage BHT, manufactured by Kawaguchi Chemical Industry Co., Ltd.) was added in place of the phenolic radical scavenger in Example 1.

As for this (P18), the content of isocyanate groups was 1.7%, the number average molecular weight (Mn) was 5,800, and the content of alkaline metals and/or alkaline earth metals was 0.03 mmol/kg.

<Example 19 (modification example 2 of a radical scavenger in Example 1)>

The medical adhesive (P19) of the present invention was obtained by the same method as that in Example 1, except that butylated hydroxyanisole (Orient BHT, manufactured by Orient Chemical Industries, Ltd.) was added in place of the phenolic radical scavenger in Example 1.

As for this (P19), the content of isocyanate groups was 1.8%, the number average molecular weight (Mn) was 5,800, and the content of alkaline metals and/or alkaline earth metals was 0.02 mmol/kg.

<Example 20 (modification example 3 of a radical

scavenger in Example 1)>

The medical adhesive (P20) of the present invention was obtained by the same method as that in Example 1, except that stearyl- β -(3,5-di-t-butyl-4-hydroxyphenyl)propionate (Adekastab AO-50, manufactured by Asahi Denka Co., Ltd.) was added in place of the phenolic radical scavenger in Example 1.

As for this (P20), the content of isocyanate groups was 1.9%, the number average molecular weight (Mn) was 5,800, and the content of alkaline metals and/or alkaline earth metals was 0.02 mmol/kg.

<Example 21 (modification example 4 of a radical scavenger in Example 1)>

The medical adhesive (P21) of the present invention was obtained by the same method as that in Example 1, except that 2,2'-methylenebis(4-ethyl-6-t-butylphenol) (Antage W-500, manufactured by Kawaguchi Chemical Industry Co., Ltd.) was added in place of the phenolic radical scavenger in Example 1.

As for this (P21), the content of isocyanate groups was 1.8%, the number average molecular weight (Mn) was 5,700, and the content of alkaline metals and/or alkaline earth metals was 0.03 mmol/kg.

<Example 22 (modification example 5 of a radical scavenger in Example 1)>

The medical adhesive (P22) of the present invention was obtained by the same method as that in Example 1, except that 4,4'-butylidenebis(3-methyl-6-t-butylphenol) (Antage Crystal, manufactured by Kawaguchi Chemical Industry Co., Ltd.) was added in place of the phenolic radical scavenger in Example 1.

As for this (P22), the content of isocyanate groups was 1.8%, the number average molecular weight (Mn) was 5,800, and the content of alkaline metals and/or alkaline earth metals was 0.02 mmol/kg.

<Example 23 (modification example 6 of a radical scavenger in Example 1)>

The medical adhesive (P23) of the present invention was obtained by the same method as that in Example 1, except that 4,4'-thiobis(3-methyl-6-t-butylphenol) (Antage W-300, manufactured by Kawaguchi Chemical Industry Co., Ltd.) was added in place of the phenolic radical scavenger in Example 1.

As for this (P23), the content of isocyanate groups was 1.8%, the number average molecular weight (Mn) was 5,800, and the content of alkaline metals and/or alkaline earth metals was 0.03 mmol/kg.

<Example 24 (modification example 7 of a radical scavenger in Example 1)>

The medical adhesive (P24) of the present invention was obtained by the same method as that in Example 1, except that 1,6-hexanediol-bis[3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate] (IRGANOX s259, manufactured by Ciba Speciality Chemicals) was added in place of the phenolic radical scavenger in Example 1.

As for this (P24), the content of isocyanate groups was 1.9%, the number average molecular weight (Mn) was 5,800, and the content of alkaline metals and/or alkaline earth metals was 0.02 mmol/kg.

<Example 25 (modification example 8 of a radical scavenger in Example 1)>

The medical adhesive (P25) of the present invention was obtained by the same method as that in Example 1, except that 3,9-bis[1,1-dimethyl-2-[β -(3-t-butyl-4-hydroxy-5-methylphenyl)propionyl]ethyl]2,4,8,10-tetraoxaspiro[[5.5]]undecane (Adekastab AO-80, manufactured by Asahi Denka Co., Ltd.) was added in place of the phenolic radical scavenger in Example 1.

As for this (P25), the content of isocyanate groups was 1.7%, the number average molecular weight (Mn) was 5,900, and the content of alkaline metals and/or alkaline earth metals was 0.02 mmol/kg.

<Example 26 (modification example 9 of a radical scavenger in Example 1)>

The medical adhesive (P26) of the present invention was obtained by the same method as that in Example 1, except that 1,3,5-trimethyl-2,4,6-tris(3,5-di-t-butyl-4-hydroxybenzyl)benzene (Adekastab AO-330, manufactured by Asahi Denka Co., Ltd.) was added in place of the phenolic radical scavenger in Example 1.

As for this (P26), the content of isocyanate groups was 1.8%, the number average molecular weight (Mn) was 5,700, and the content of alkaline metals and/or alkaline earth metals was 0.03 mmol/kg.

<Example 27 (modification example 10 of a radical scavenger in Example 1)>

The medical adhesive (P27) of the present invention was obtained by the same method as that in Example 1, except that bis[3,3'-bis-(4'-hydroxy-3'-t-butylphenyl)butylic acid]glycol ester (Antioxidant TMOZ, manufactured by Hoechst AG) was added in place of the phenolic radical scavenger in Example 1.

As for this (P27), the content of isocyanate groups was 1.9%, the number average molecular weight (Mn) was 5,900, and the content of alkaline metals and/or alkaline earth metals was 0.02 mmol/kg.

Results

The medical adhesives (P16) to (P27) were evaluated by the procedures employed in Evaluations 1 and 2 of the specification of the present application. The results are shown in Tables 1-1 and 1-2.

Table 1-1

	Example					
	16	17	18	19	20	21
Change in appearance	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent
Wet adhesive strength (2H)	1.4	1.3	1.2	1.2	1.1	1.2
Wet adhesive strength (5D)	1.3	1.4	1.1	1.2	1.2	1.2

Table 1-2

	Example					
	22	23	24	25	26	27
Change in appearance	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent
Wet adhesive strength (2H)	1.2	1.2	1.2	1.1	1.2	1.2
Wet adhesive strength (5D)	1.1	1.2	1.3	1.2	1.3	1.1

Conclusion

From the results shown above, the following facts 1 to 2 can be clearly read.

1. When the fluorine-containing aliphatic diisocyanate (A11) in Example 1 is modified to compounds in Examples 16 and 17 respectively, Change in appearance, Wet adhesive strength (2H), and Wet adhesive strength (5D) are

comparable before and after substitution. The effect of the present invention can be exerted.

2. When the phenolic radical scavenger (PRS) in Example 1 is modified to compounds in Examples 18 to 27 respectively, Change in appearance, Wet adhesive strength (2H), and Wet adhesive strength (5D) are comparable before and after substitution. The effect of the present invention can be exerted.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Signed this *20th* day of ~~*20th*~~ *August*, 2009
T.Y.

Tetsuji Yoshimura

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